PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY
(Chapter I of the Patent Cooperation Treaty)

(PCT Rule 44*bis*)

Applicant's or agent's file reference P08498PCT	**FOR FURTHER ACTION**	See item 4 below
International application No. PCT/US2005/009763	International filing date *(day/month/year)* 24 March 2005 (24.03.2005)	Priority date *(day/month/year)* 24 March 2004 (24.03.2004)
International Patent Classification (8th edition unless older edition indicated) See relevant information in Form PCT/ISA/237		
Applicant H. C. STARCK INC.		

1. This international preliminary report on patentability (Chapter I) is issued by the International Bureau on behalf of the International Searching Authority under Rule 44 *bis*.1(a).

2. This REPORT consists of a total of 11 sheets, including this cover sheet.

   In the attached sheets, any reference to the written opinion of the International Searching Authority should be read as a reference to the international preliminary report on patentability (Chapter I) instead.

3. This report contains indications relating to the following items:

☒	Box No. I	Basis of the report
☐	Box No. II	Priority
☒	Box No. III	Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
☒	Box No. IV	Lack of unity of invention
☒	Box No. V	Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
☐	Box No. VI	Certain documents cited
☐	Box No. VII	Certain defects in the international application
☒	Box No. VIII	Certain observations on the international application

4. The International Bureau will communicate this report to designated Offices in accordance with Rules 44*bis*.3(c) and 93*bis*.1 but not, except where the applicant makes an express request under Article 23(2), before the expiration of 30 months from the priority date (Rule 44*bis* .2).

	Date of issuance of this report 26 September 2006 (26.09.2006)
The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland Facsimile No. +41 22 338 82 70	Authorized officer Dorothée Mülhausen e-mail: pt01@wipo.int

Form PCT/IB/373 (January 2004)

PATENT COOPERATION TREATY

From the
INTERNATIONAL SEARCHING AUTHORITY

To:

see form PCT/ISA/220

WRITTEN OPINION OF THE INTERNATIONAL SEARCHING AUTHORITY
(PCT Rule 43*bis*.1)

Date of mailing
(day/month/year) see form PCT/ISA/210 (second sheet)

Applicant's or agent's file reference
see form PCT/ISA/220

FOR FURTHER ACTION
See paragraph 2 below

International application No.	International filing date *(day/month/year)*	Priority date *(day/month/year)*
PCT/US2005/009763	24.03.2005	24.03.2004

International Patent Classification (IPC) or both national classification and IPC
B81C1/00, C23C16/06, C30B23/00, C30B25/00, C23C14/16, C23C14/54, C30B29/02

Applicant
H.C. STARCK INC.

1. This opinion contains indications relating to the following items:

- ☒ Box No. I Basis of the opinion
- ☐ Box No. II Priority
- ☒ Box No. III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- ☒ Box No. IV Lack of unity of invention
- ☒ Box No. V Reasoned statement under Rule 43*bis*.1(a)(i) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- ☐ Box No. VI Certain documents cited
- ☐ Box No. VII Certain defects in the international application
- ☒ Box No. VIII Certain observations on the international application

2. **FURTHER ACTION**

If a demand for international preliminary examination is made, this opinion will usually be considered to be a written opinion of the International Preliminary Examining Authority ("IPEA"). However, this does not apply where the applicant chooses an Authority other than this one to be the IPEA and the chosen IPEA has notifed the International Bureau under Rule 66.1*bis*(b) that written opinions of this International Searching Authority will not be so considered.

If this opinion is, as provided above, considered to be a written opinion of the IPEA, the applicant is invited to submit to the IPEA a written reply together, where appropriate, with amendments, before the expiration of three months from the date of mailing of Form PCT/ISA/220 or before the expiration of 22 months from the priority date, whichever expires later.

For further options, see Form PCT/ISA/220.

3. For further details, see notes to Form PCT/ISA/220.

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WRITTEN OPINION OF THE INTERNATIONAL SEARCHING AUTHORITY

International application No.
PCT/US2005/009763

Box No. I Basis of the opinion

1. With regard to the **language**, this opinion has been established on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.

   ☐ This opinion has been established on the basis of a translation from the original language into the following language , which is the language of a translation furnished for the purposes of international search (under Rules 12.3 and 23.1(b)).

2. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application and necessary to the claimed invention, this opinion has been established on the basis of:

   a. type of material:

   ☐ a sequence listing

   ☐ table(s) related to the sequence listing

   b. format of material:

   ☐ in written format

   ☐ in computer readable form

   c. time of filing/furnishing:

   ☐ contained in the international application as filed.

   ☐ filed together with the international application in computer readable form.

   ☐ furnished subsequently to this Authority for the purposes of search.

3. ☐ In addition, in the case that more than one version or copy of a sequence listing and/or table relating thereto has been filed or furnished, the required statements that the information in the subsequent or additional copies is identical to that in the application as filed or does not go beyond the application as filed, as appropriate, were furnished.

4. Additional comments:

WRITTEN OPINION OF THE INTERNATIONAL SEARCHING AUTHORITY

International application No.
PCT/US2005/009763

Box No. III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability

The questions whether the claimed invention appears to be novel, to involve an inventive step (to be non obvious), or to be industrially applicable have not been examined in respect of:

☐ the entire international application,

☒ claims Nos. 5-16, 19-23, 25-28

because:

☐ the said international application, or the said claims Nos. relate to the following subject matter which does not require an international preliminary examination *(specify)*:

☐ the description, claims or drawings *(indicate particular elements below)* or said claims Nos. are so unclear that no meaningful opinion could be formed *(specify)*:

☐ the claims, or said claims Nos. are so inadequately supported by the description that no meaningful opinion could be formed.

☒ no international search report has been established for the whole application or for said claims Nos. 5-16, 19-23, 25-28

☐ the nucleotide and/or amino acid sequence listing does not comply with the standard provided for in Annex C of the Administrative Instructions in that:

the written form	☐	has not been furnished
	☐	does not comply with the standard
the computer readable form	☐	has not been furnished
	☐	does not comply with the standard

☐ the tables related to the nucleotide and/or amino acid sequence listing, if in computer readable form only, do not comply with the technical requirements provided for in Annex C-*bis* of the Administrative Instructions.

☐ See separate sheet for further details

Box No. IV Lack of unity of invention

1. ☒ In response to the invitation (Form PCT/ISA/206) to pay additional fees, the applicant has:

   ☐ paid additional fees.

   ☐ paid additional fees under protest.

   ☒ not paid additional fees.

2. ☐ This Authority found that the requirement of unity of invention is not complied with and chose not to invite the applicant to pay additional fees.

3. This Authority considers that the requirement of unity of invention in accordance with Rule 13.1, 13.2 and 13.3 is

   ☐ complied with

   ☒ not complied with for the following reasons:

   see separate sheet

4. Consequently, this report has been established in respect of the following parts of the international application:

   ☐ all parts.

   ☒ the parts relating to claims Nos. 1-4,17,18,24

Box No. V Reasoned statement under Rule 43*bis*.1(a)(i) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims	
	No:	Claims	1-4,17,18,24
Inventive step (IS)	Yes:	Claims	
	No:	Claims	1-4,17,18,24
Industrial applicability (IA)	Yes:	Claims	1-4,17,18,24
	No:	Claims	

2. Citations and explanations

see separate sheet

Box No. VIII Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:

see separate sheet

Re Item IV.

1. This Authority considers that there are 4 inventions covered by the claims indicated as follows:

   I. Claims 1 to 4, 17, 18 and 24 are directed to a tantalum film with a **nanocrystalline microstructure**, method for forming the nanocrystalline Ta film and a device comprising the nanocrystalline Ta film.

   II Claims 5 to 8, 19, 20, 25 and 26 are directed to a tantalum film with a **single crystal microstructure**, method for forming the single crystal Ta film and a device comprising the single crystal Ta film.

   III Claims 9 to 11, 21 to 23, 27 and 28 are directed to a tantalum film with an **amorphous microstructure**, method for forming the amorphous Ta film and a device comprising the amorphous Ta film.

   IV Claims 12 to 16 are directed to a depositing method for forming a tantalum film with **any microstructure**.

   In conclusion, the groups of claims are not linked by common or corresponding special technical features and define 4 different inventions not linked by a single general inventive concept.
   The application, hence does not meet the requirements of unity of invention as defined in Rules 13.1 and 13.2 PCT.

Re Item V.

1. Reference is made to the following document:

   D1= WO 98/54377 A (APPLIED MATERIALS, INC; CHIANG, TONY; DING, PEIJUN; CHIN, BARRY, L; SU) 3 December 1998 (1998-12-03);

   D2= GE S H ET AL: "Structures and magnetic properties of Co/Ta multilayered thin films" PHYSICA STATUS SOLIDI A GERMANY, vol. 132, no. 2, 16 August 1992 (1992-08-16), pages 487-493, XP009052667 ISSN: 0031-8965;

   D3= PARFITT L J ET AL: "ORIGINS OF RESIDUAL STRESS IN MO AND TA

FILMS: THE ROLE OF IMPURITIES, MICROSTRUCTURAL EVOLUTION AND PHASE TRANSFORMATIONS" MATERIALS RESEARCH SOCIETY SYMPOSIUM PROCEEDINGS, MATERIALS RESEARCH SOCIETY, PITTSBURG, PA, US, vol. 436, 8 April 1996 (1996-04-08), pages 505-510, XP002074085 ISSN: 0272-9172;

D4= FRENCH B L ET AL: "Correlation of stress and phase evolution in thin Ta films on Si (100) during thermal testing" SURFACE ENGINEERING 2002 - SYNTHESIS, CHARACTERIZATION AND APPLICATIONS. SYMPOSIUM (MATER. RES. SOC. SYMPOSIUM PROCEEDINGS VOL.750) MATER. RES. SOC WARRENDALE, PA, USA, 2003, pages 367-372, XP002341641 ISBN: 1-55899-687-7;

D5= HOOGEVEEN R ET AL: "TEXTURE AND PHASE TRANSFORMATION OF SPUTTER-DEPOSITED METASTABLE TAFILMS AND TA/CU MULTILAYERS" THIN SOLID FILMS, ELSEVIER-SEQUOIA S.A. LAUSANNE, CH, vol. 275, no. 1/2, 1 April 1996 (1996-04-01), pages 203-206, XP000626342 ISSN: 0040-6090;

D6= KIM H ET AL: "THE GROWTH OF TANTALUM THIN FILMS BY PLASMA-ENHANCED ATOMIC LAYER DEPOSITION AND DIFFUSION BARRIER PROPERTIES" MATERIALS RESEARCH SOCIETY SYMPOSIUM PROCEEDINGS, MATERIALS RESEARCH SOCIETY, PITTSBURG, PA, US, vol. 716, 2002, pages 407-412, XP008026978 ISSN: 0272-9172;

D7= PINTO R ET AL: "GETTER-BIAS SPUTTERING OF HIGH PURITY METAL FILMS IN A HIGH CURRENT VACUUM DISCHARGE IN THE 10-4 TORR RANGE" JAPANESE JOURNAL OF APPLIED PHYSICS, PUBLICATION OFFICE JAPANESE JOURNAL OF APPLIED PHYSICS. TOKYO, JP, vol. 9, no. 2, February 1970 (1970-02), pages 174-181, XP000861704 ISSN: 0021-4922;

1.1. D1 discloses a method of sputter depositing tantalum films for semiconductor interconnect structures, wherein the substrate temperature during said Ta film deposition is selected to be within a range of about 100°C to about 220°C (claim 52) and at processing pressures (figure 1) in the range of 10 to 60 mTorr (1.3×10^{-5} to 8×10^{-5} bar).

The method disclosed in D1 fall within the method claimed in claim 17 and therefore, the resulting tantalum film has identical characteristics as the tantalum film claimed.

Consequently, the present application does not meet the criteria of Article 33(1) PCT, because the subject-matter of claims 1 to 4, 17 and 24 is not new in the sense of Article 33(2) PCT.

1.2. D2 discloses a method of sputter depositing tantalum films for magnetic recording material. The tantalum films show a broad X-ray diffraction peak at $2\Theta=38°$ (figure 2(b)).

The feature of a broad X-ray diffraction peak at $2\Theta=38°$ is an indication of a tantalum film having a nanocrystalline structure.
Although D2 is silent about continuous electron diffraction rings, the tantalum films of D2 would show such a ring pattern when measured.

Furthermore, the substrate temperature during the sputtering of tantalum films is not disclosed in D2. However, the substrate temperature should be in the range of 100 °C to 200 °C, because the resulting tantalum film has a nanocrystalline structure as is shown by the X-ray diffraction peak at $2\Theta=38°$.

Consequently, the present application does not meet the criteria of Article 33(1) PCT, because the subject-matter of claims 1 to 4, 17 and 24 is not new in the sense of Article 33(2) PCT.

1.3. D3 discloses nano-crystalline tantalum films for transistors obtained by magnetron sputter deposition at base pressures $< 3x10^{-6}$ Torr.
The resulting tantalum film has a nanocrystalline structure and should show, when measured, a X-ray diffraction peak at $2\Theta=38°$ and a resistance of 30 to 50 μΩ cm.

Although D3 is silent about the substrate temperature, one can reasonably assume that the substrate temperature during deposition falls within the range of 100 °C to 200 °C, because the resulting tantalum film has a nanocrystalline structure.

Consequently, the present application does not meet the criteria of Article 33(1) PCT, because the subject-matter of claims 1 to 4, 17, 18 and 24 is not new in the sense of Article 33(2) PCT.

1.4. D4 discloses the dc magnetron sputter deposition of tantalum films at a base pressure less than 10^{-7} Torr. The as-deposited tantalum film (figure 3b) is characterised by a broad X-ray diffraction peak at $2\Theta=38°$.

The broad X-ray diffraction peak at $2\Theta=38°$ is a characteristic of the nanocrystalline microstructure, i.e. the tantalum film has a nanocrystalline microstructure.

Although D4 is silent about the substrate temperature, one can reasonably assume that the substrate temperature during deposition falls within the range of 100°C to 200°C, because the resulting tantalum film has a nanocrystalline structure.

Although D4 is silent about continuous electron diffraction rings, the tantalum films of D4 would show such a ring pattern when measured.

Consequently, the present application does not meet the criteria of Article 33(1) PCT, because the subject-matter of claims 1, 3, 4, 17 and 24 is not new in the sense of Article 33(2) PCT.

1.5. D5 discloses the dc magnetron sputter deposition of tantalum films on silicon substrates at 100°C at a base pressure less than 10^{-7} mTorr.

The method disclosed in D5 fall within the method claimed in claim 17 and therefore, the resulting tantalum film has identical characteristics as the tantalum film claimed.

Consequently, the present application does not meet the criteria of Article 33(1) PCT, because the subject-matter of claims 1, 3, 4, 17 and 24 is not new in the sense of Article 33(2) PCT.

1.6. D6 discloses the deposition of tantalum films by plasma-enhanced atomic layer deposition from room temperature to 300°C. The tantalum films are composed of

nano-grains and show a broad X-ray diffraction peak at $2\Theta=38°$ (figure 1).

Although D6 is silent about continuous electron diffraction rings, the tantalum films of D6 would show such a ring pattern when measured.

Consequently, the present application does not meet the criteria of Article 33(1) PCT, because the subject-matter of claims 1, 3, 4, 17 and 24 is not new in the sense of Article 33(2) PCT.

1.7. D7 discloses a method of sputter depositing tantalum films for semiconductor interconnect structures, wherein the substrate temperature during said Ta film deposition is 140°C and the processing pressure is 3.6×10^{-4} Torr (4.8×10^{-7} bar).

The method disclosed in D7 falls within the method claimed in claim 17 and therefore, the tantalum film obtained by the method of D7 should have has identical characteristics as the tantalum film claimed in the present application.

Consequently, the present application does not meet the criteria of Article 33(1) PCT, because the subject-matter of claims 1 to 4, 17 and 24 is not new in the sense of Article 33(2) PCT.

Re Item VIII.

1. The application does not meet the requirements of Article 6 PCT, because claims 1, 4, 17 and 18 are not clear.

1.1. Claims 4 and 17 do not meet the requirements of Article 6 PCT in that the matter for which protection is sought is not clearly defined.
The claims attempt to define the subject-matter in terms of the result to be achieved, which merely amounts to a statement of the underlying problem, without providing the technical features necessary for achieving this result.

1.2. The relative term "broad" used in claim 1 has no well-recognised meaning and leaves

the reader in doubt as to the meaning of the technical feature to which it refers, thereby rendering the definition of the subject-matter of claim 1 unclear, Article 6 PCT.

1.3. The unit "Torr" employed in claim 18 and throughout the description is not additionally expressed in terms of the units stipulated by Rule 10.1/(a) PCT.